NIST PRESSURE CELL ASSEMBLY INSTRUCTIONS

GENERAL RULES

- 1. ALWAYS wear safety glasses when working with the pressure cell. No exceptions.
- 2. NEVER look directly into the ends of the cell when it is under pressure. Use a flashlight and a mirror to inspect the windows and your sample.
- 3. ALWAYS use brass, wood, or plastic tools when working near the windows or the steel cell body. ALWAYS push against just the o-ring. NEVER push or pry against the windows or the cell body. Steel tools will scratch the windows or the cell body, and will permanently damage the cell.
- 4. CLEAN, CLEAN Have a clean workspace and lint-free towels at hand. The sealing surfaces must not be marred or dirty.

FOR SAMPLES USING THE TEFLON SLEEVE INSERTS

- 1. Set the cell body on one of the end caps, but don't bolt anything down.
- 2. Insert one backer o-ring into the cell body. Push it down until it meets the support face of the end cap. The backer ring should have the cupped side facing up.
- 3. Insert one o-ring on top of the backer ring.
- 4. Insert the Teflon sleeve (one with a 1-mm inner lip) into the body, and push it down with an extra (steel) sleeve until it is fully seated against the two o-rings.
- 5. Put an o-ring around one of the sapphire windows, about one-third of the way from one end. Gently push the window into the Teflon sleeve. The o-ring will ride at the top of the cell body. Alternately push the window down and tuck the o-ring into the groove with the brass tools, being careful not to nick the window. The o-ring should be pulled into the body with the window. Once it is below the top surface of the body and below the bevel of the window, a steel sleeve can be used to seat the o-ring.
- 6. Insert a backer ring on top of the first o-ring, with the cupped side down. Use the brass tools to push it down, past the bevel on the window.
- 7. Put the second end cap on (gently) since the window may not be fully seated yet. Be sure the alignment marks between the body and end cap are matched. If the end cap does not initially seat properly, don't force it tighten the bolts slowly in a cross pattern with a minimum of pressure to seat the window evenly. Bolt the end cap down in a cross pattern. Just snug the bolts at this point. Do not torque them down yet.
- 8. Pick up the assembled end cap and body, remove the unattached end cap, and flip the assembly over.
- 9. Remove the two o-rings on this side. Use a steel sleeve to insure that the Teflon sleeve is fully seated in the direction of the assembled end.
- 10. It is now time to add the sample. Fill a syringe with approximately 5 ml of sample, free from bubbles. Fill the Teflon sleeve with the sample until there is a bulging meniscus at the top of the sleeve. Gently remove any air bubbles from the interior of the sleeve with the tip of the syringe.

- 11. Put an o-ring near the top-third of the second window. Touch the face of the window to the meniscus, being sure there are no trapped bubbles. Slowly insert the window into the sleeve. Excess sample will be expelled from the thin gap between the window and the sleeve. Alternately push the window and blot the sample until the window is approximately two-thirds of the way in. At this point, the o-ring should be touching the cell body. Continue pushing and blotting, and now tuck the o-ring into the groove as well, leaving an escape for the liquid (careful it may squirt out). Once the window is seated, force the o-ring completely into the body.
- 12. Check for air bubbles in the sample. If bubbles are present, you'll have to start over. Wrap a thick towel around the end of a nutdriver (NOT a screwdriver) to protect the window, and force the window and sleeve out of the body from the assembled and. Go back to step #1. If there are no bubbles in your sample, proceed.
- 13. Put the backer ring onto the o-ring with the cupped side facing the o-ring, and push it down with the brass tools.
- 14. Put the second end cap on (gently) since this window will probably not be fully seated yet. If the end cap does not initially seat properly, don't force it tighten the bolts slowly in a cross pattern with a minimum of pressure to seat the window evenly. Hand-tighten the end cap down in a cross pattern.
- 15. Torque the bolts down in a three-step process. Always use the torque wrench, and tighten the bolts in a cross pattern. Alternate between ends of the cell. The torque specifications for the three steps are 8 ft-lb., 16 ft-lb., then 25 ft-lb., respectively. (96 in-lb., 192 in-lb., and 300 in-lb.)
- 16. Now it is necessary to seat the o-rings towards the end caps by pressurizing the sample with nitrogen.

FOR SAMPLES USING THE STEEL SLEEVE INSERTS

- 1. Set the cell body on one of the end caps, but don't bolt anything down.
- 2. Insert one backer o-ring into the cell body. Push it down until it meets the support face of the end cap. The backer ring should have the cupped side facing up.
- 3. Insert one o-ring on top of the backer ring.
- 4. Insert the steel sleeve (one with a 1-mm inner lip) into the body, and push it down with an extra (steel) sleeve until it is fully seated against the two o-rings.
- 5. Put an o-ring around one of the sapphire windows, about one-third of the way from one end. Gently slide the window into the steel sleeve. Don't force it, or you will chip the window. The o-ring will ride at the top of the cell body. Alternately push the window down and tuck the o-ring into the groove with the brass tools, being careful not to nick the window. The o-ring should be pulled into the body with the window. Once it is below the top surface of the body and below the bevel of the window, a steel sleeve can be used to seat the o-ring.
- 6. Insert a backer ring on top of the first o-ring, with the cupped side down. Use the brass tools to push it down, past the bevel on the window.
- 7. Put the second end cap on (gently) since the window may not be fully seated yet. Be sure the alignment marks between the body and end cap are matched. If the end cap does not initially seat properly, don't force it tighten the bolts slowly in a cross pattern with a minimum of

- pressure to seat the window evenly. Bolt the end cap down in a cross pattern. Just snug the bolts at this point. Do not torque them down yet.
- 8. Pick up the assembled end cap and body, remove the unattached end cap, and flip the assembly over.
- 9. Remove the two o-rings on this side. Use a second sleeve to insure that the steel sleeve is fully seated in the direction of the assembled end. IMPORTANT! Rotate the sleeve to line up the four holes in the sleeve with the four ports on the body.
- 10. A pressed polymer sample mounted in an o-ring can be placed in the cell now, or a liquid sample will be purged through the entire cell once fully assembled.
- 11. Put an o-ring near the middle of the second window. Slowly insert the window into the metal sleeve. Then alternately push the window down and tuck the o-ring into the body using the brass tools. Seat the o-ring with an extra sleeve.
- 12. Put the backer ring onto the o-ring with the cupped side facing the o-ring, and push it down with the brass tools.
- 13. Put the second end cap on (gently) since this window will probably not be fully seated yet. If the end cap does not initially seat properly, don't force it tighten the bolts slowly in a cross pattern with a minimum of pressure to seat the window evenly. Hand-tighten the end cap down in a cross pattern.
- 14. Torque the bolts down in a three-step process. Always use the torque wrench, and tighten the bolts in a cross pattern. Alternate between ends of the cell. The torque specifications for the three steps are 8 ft-lb., 16 ft-lb., then 25 ft-lb., respectively. (96 in-lb., 192 in-lb., and 300 in-lb.)
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SEATING THE WINDOWS WITH NITROGEN

- 1. Completely assemble the cell, tightening both end caps to the specified torque.
- 2. Fit the cell body with three plugs and a single valve (60k psig).
- 3. Connect the high outlet pressure regulator to the nitrogen tank. Be sure the regulator is closed, and open the tank.
- 4. Connect the AF1 (1/16") line to the regulator and to the valve that is fitted to the cell.
- 5. With the valve to the cell open, use the regulator to slowly increase the pressure in the line and cell to approximately 500 psig.
- 6. Close the valve closest to the cell, to isolate the cell.
- 7. Close the valve on the nitrogen tank.
- 8. Carefully crack the AF1 fitting near the cell to release the pressure in the line. This should be a very small volume of nitrogen released.
- 9. Crack the smaller valve near the regulator to vent the pressure from the regulator.
- 10. Disconnect the AF1 line from the pressure cell.
- 11. Allow approximately 5 minutes for the o-ring seals to seat, then vent the cell, while listening for the (very small) rush of air, indicating that the cell was holding pressure. If no air was released, there is a leak that must be found before continuing.
- 12. If the cell held compressed air, the windows and o-rings should now be fully seated.

ASSEMBLY OF 5 MM PATHLENGTH CELL

- 1. Use a steel or Teflon sleeve with 5 mm inner lip. These sleeves are slightly longer than the 1 mm sleeves.
- 2. To accommodate the longer sleeve, (2)-2 mm spacer plates must be added to extend the length of the center body, adding one plate to each side. In the first step, place a spacer on the bottom end cap before the cell body. Once seated, the window and backer o-ring will be nearly flush with the top surface of the cell body. Place the second spacer plate on top of the body before bolting down the end cap.
- 3. Other assembly is identical to the instructions above for the Teflon or steel sleeves.

DISASSEMBLY OF THE CELL

- 4. Be sure that the pressure has been fully released from the cell before disassembly.
- 5. Loosen bolts from both end caps, but do not fully remove the bolts.
- 6. With the cell sitting on one end, remove all of the bolts from the end cap. Lift the end cap from the body, if possible. The end cap will likely be stuck, and can be gently lifted using two 1/4-28 bolts in the threaded holes in the end caps. Insert the two bolts just until they touch the body. Tighten each bolt 1/8 turn, alternating to lift the cap straight up until it is free.
- 7. Flip the cell over, and remove the second end cap in the same manner.
- 8. With both end caps removed, push the windows and sleeve from the body of the cell. Firm pressure with the thumbs is sufficient. No metal tools!
- 9. With the windows and sleeve out, clean and dry everything.

MISCELLANEOUS NOTES

- 1. The path length of the cells increases slightly as a function of pressure. The increase is most significant when using the 1 mm path length, which increases to approximately 1.1mm at 45,000 psig. Check the logbook for the actual pathlength.
- 2. Coat the bolts occasionally with anti-seize compound.
- 3. Pressure cell O-rings are size 116 (aerospace designation AS568A-116. They are 3/32" wide, 3/4"ID and 15/16" OD. Viton, ethylene-propylene and silicone are the most commonly used materials. Teflon encapsulated silicone can also be used. Piston o-rings are size 003 (1/16" wide, 1/16" ID, 3/16" OD).

TORQUE SPECIFICATIONS

These are the minimum torque specifications, as recommended by HiP:

Low pressure **AF1** fittings (15,000 psig, 1/16" tubing): 55 in-lbs. = 4.5 ft-lbs.

Medium pressure **HF2** fittings (30,000 psig, 1/8" tubing): 75 in-lbs. = 6.3 ft-lbs.

High pressure **HF4** fittings (60,000 psig, 1/4" tubing): 25 ft-lbs.



